US ERA ARCHIVE DOCUMENT

Magnitude of the Residue **OPPTS 860.1500** DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043



# UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460

PREVENTION, PESTICIDES AND TOXIC SUBSTANCES

# **MEMORANDUM**

Date:

July 2, 2003

Reviewers:

Date: 9.2.03

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DP Barcode: D278386

Petition No.: PP#1F06313

Citation:

45405118 Versoi, P.; Abdel-Baky, S. (2000) The Magnitude of BAS 510 F

Residues in Red Raspberries and Highbush Blueberries: Final Report: Lab Project Number: 2000/5195: 63912: 99277/NY/1. Unpublished study prepared by BASF.

Corporation. 53 p.

Sponsor:

**BASF** Corporation

#### Background

The information contained herein was compiled by Dynamac Corporation (20440 Century Boulevard, Suite 100, Germantown MD 20874), contractor, under the supervision of RAB2/HED. This DER has undergone secondary review by RAB2, and reflects current HED and Office of Pesticide Programs (OPP) policies. This DER was also peer reviewed by PMRA.

#### **Executive Summary**

BASF Corporation has submitted field trial data on the representative crops (highbush blueberry and red raspberry) of the berry crop group (crop group 13). Six blueberry trials were conducted in Regions 1 (1 trial; NY), 2 (2 trials; GA), 5 (2 trials; WI), and 12 (1 trial; OR) and three raspberry trials were conducted in Regions 1 (1 trial; NY) and 12 (2 trials; OR). The number and

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

location of field trials satisfy the US EPA's data requirements with respect to the geographic representation of residue data for the berries crop group.

For both high bush and low bush blueberries, the PMRA's data requirements have not been met. Additional trials in both zones 1A (3 trials) and 5A (3 trials) are required by the PMRA to support a national registration for blueberries. The PMRA can, however, support a registration for high bush blue berries with the data provided. For raspberries, additional trials (one additional in each of zones 5 and 5B) will be required as a condition of registration in Canada.

At each test location, the 70% WG formulation of BAS 510 F was applied four times as a foliar spray at ~0.37 lb ai/A/application, (0.414 ka ai/ha/application) with a 6- to 9-day retreatment interval, for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha/season). Mature samples were collected at a 0-day post-treatment interval. In one raspberry field trial, additional samples were collected at 2, 4, 6, and 8 days following treatment to evaluate residue decline.

Residues of BAS 510 F in/on blueberries and raspberries were quantitated using a validated LC/MS/MS method (D9908), the data collection method for plant commodities. Storage stability data (refer to the DER for MRID 45405109) are available to support the 96 days storage interval for the samples from the submitted berry field trials.

At the applied total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha/season), the ranges of BAS 510F residues in/on treated mature samples were 0.49-2.50 ppm for blueberries and 1.39-3.31 ppm for raspberries. The residue decline data generated in New York State for raspberries indicated that residues of BAS 510 F decreased at longer post-treatment intervals with a half-life of approximately 8 days and complete dissipation anticipated by 15 days.

Residue data from the current submission are acceptable to fulfill the US EPA's crop field trial data requirements by this use pattern for the berry crop group (crop group 13). Sufficient data are available for the PMRA to support a registration on high bush blueberries and a registration on the remaining crops within this crop group, provided additional reside trials in raspberries (one trial in each of zones 5 and 5A) are carried out and submitted as a condition of registration.

## **GLP Compliance**

Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported which would impact the study results or their interpretation.

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

# 1. Materials and Methods

## 1.1. Test Substance

# Active Ingredient

Common Name: Nicobifen (ISO, proposed)

IUPAC Name: 2-Chloro-N-(4'-chlorobiphenyl-2-yl)nicotinamide

CAS Name: 3-Pyridinecarboxamide, 2-chloro-N-(4'chloro[1,1'-biphenyl]-2-yl)-

CAS Number: 188425-85-6 Company Name: BAS 510 F

Other Synonyms: BASF Registry No. 300355

#### Structure:

BAS 510 F

T/	ABLE B.	1.2.Tria	Numbe	rs and G	eograpi	nical Lo	cations	
		Raspbe	rry, red¹			Bluel	erries	***************************************
NAFTA Growing	Car	nada	ι	JS	Car	nada	τ	S
Region	SUB	REQ	SUB	REQ <sup>2</sup>	SUB	REQ	SUB	REQ
1	1		1		1	1	1	1
1A			*			3		
2					2		2	2
3								
4								
5		1			2		2	2
5A						3		
5B		1						
6								
7								
7A								
8								
9								
10								
11								
12	2	3	2 -			1		
13								
14							÷	
15								
16								
17								
18								
19								
20								
21					1			***************************************
Total	3	5	3	N/A	5	8	5	5

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

<sup>1</sup> The representative commodities for berries crop group are any one blackberry or any one raspberry and highbush blueberry.

<sup>&</sup>lt;sup>2</sup> OPPTS 860.1500 Table 5 does not identify specific regions for the reduced number of raspberry field trials required as a representative crop of the berries crop group. However, trials were conducted for raspberries in Regions 1 and 12, which together account for 82% of raspberry production (OPPTS 860.1500, Table 6).

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

Region (County, Staty Year)  Blueberry, highbush	100		100000000000000000000000000000000000000								
Blueberry,	Region (County, State,	Crop, Variety	Formul.	Applic. Timing	Applic. Rate (1b ai/A) [kg ai/ha]	Retreat. Intervals (days)	No. of Applics.	Applic. Method/ Applic. Volume (GPA) [l/ha]	Total Applic. Rate (ib ai/A) [kg ai/ha]	Tank Mix Adjuvants	Harvest Procedures
2	highbush										
<u> </u>	Yates, NY, 1999	Highbush blueberry,	70% WG	Verasion (coloration)	0.37-0.39 [0.414-	6-7	4	Foliar spray/ 60.3-62.6	1.52	Activate Plus (0.0025%,	Berries harvested 0 days after last application
		Blue Ray and Blue Cron		early maturity	0.437.]			[102-529]		(A:'A	(DALA).
				early maturity							
				mature berries							
2 Triff	Tift, GA, 1999	Highbush	70% WG	1% ripe fruit	0.367	۲	4	Foliar spray/	1.475	Latron	Berries harvested 0
		blueberry, Tiff Blue		10% ripe fruit	0.372	•		53.3-56.8	[1.652]	B-1956	DALA.
				30% ripe fruit	0.417]					) in the second	
				50% ripe fruit							
7	Colquitt, GA,	Highbush	70% WG	40% ripe fruit	0.364	7	4	Foliar spray/	1.498	Latron	Berries harvested 0
Ž	2	blueberry, Climax		70% ripe fruit	0.381			543-57.0	[1.678]	B-1956	DALA.
				95% ripe fruit	0.427]						
				mature fruit							
5 Pie	Pience, WI,	Highbush	70% WG	0.25" diameter	0.37	6-7	4	Foliar spray/	1.48	X77	Berries harvested 0
866 	2	Slite Chary,		0.5" diameter	[0.414]			49.6-50.43	[1.658]	(602/100gal)	DALA.
				20% ripe							
				85% ripe							
20 1	Jackson, WI,		70% WG	0.25" diameter	0.37-0.383	6-9	4	Foliar spray/	1.513	LEX	Berries harvested 0
	2	blueberry. Berkley		0.5" diameter	0.414			50.5-51.7	[1.694]	(602/100gal)	DALA.

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

	(40z/100gal)	S.	[609-642]			0.43]			Lulamene		
DAIA	98	[1.67]	54.42.57.3			0.384	THE		raspberry,	OK. 1999	
Berries harvested 0	Triton AG	1.4885	Foliar spray/	4	7	0.365	Bloom - green	70% WG	REA	Washington,	12
											L
			William (n.				mature fruit				-
		na weedinii				-	green, red fruit	icana			
		*					HUI	•			
	(TOB LOOKAL)					0.433]	Bloom - green		*	-	
Ş	A ye	[50.1]	K 1 / / / / / / / / / / / / / / / / / /			D45.			Mocker		
Berries harvested 0	Triton AG	1.4996	Foliar spray/	4	7	0.368	Bloom - green	70% WG	7 R	Washington, OR 1999	IJ
							mature berries				
							0.625" berries			-	
	Ş		[009-707]			S. Đ	0.5" berries		Î		
o, and a DALA.	(0.0025%,	[40.1]	1.50-7.76				ocu ica		Tital	Contine shidy	
Berries harvested 0, 2, 4,	Activate Plus		Foliar spray/	4	0	0.37.0.39		70% WG	Red	Togs, NY,	<b>}</b>
		÷	-							77, 724	Raspberry, red
-							mature		·		
							50% colored				
	(30z/100gal)		[1131-1142]			0.454	10% ripe		bille Crop		
DALA	B-1956	[1.684]	101.08-102.02			0.379	cluster	2	blueberry.	Š	
Berries harvested 0	Latron	1.504	Foliar spray/	٨	£	0.375-	1-2 barries in	70% WG	Highbush	Benton, OR,	12
						a de la companya de	80% ripe				
							70% ripe				
			[l/ha]			3					
		(FailA)	Applic.		(days)				-	<u> </u>	
	Adjuvants	Rate	Method/	Applics.	Intervals	Rate				(County, State,	Region
Harvest Procedures	Tank Mix	Total Applic.	Applic.	No. of	Retreat.	Applic.	Applic. Timing	Formul.	Crop, Variety	Location	EP/A
^					÷			<b>Bion</b>	eld Trial Inform	Table 1.2.2. Crop and Field Trial Information	Table 1:

Berry Crop Group PMRA a.i. code (CCH) BAS 510 F

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

Table 1	Table 1.2.2. Crop and Field Trial Information.	eld Trial Inform	nation.									
EPA Region	EPA Location Region (County, State, Year)	Crop, Variety Formul. Applic. 7	Formul.	Applic. Timing	Fiming Applic.  Rate (Ib ai/A) [kg ai/ha]	Retreat. No. of Intervals Applics. (days)	No. of Applics.	Applic. Method/ Applic. /olume (GPA)	Total Applic. Rate (lb ai/A) [kg ai/ha]	Tank Mix Adjuvants	Harvest Procedures	
				Bloom - green fruit								7
	-			green, red fruit mature fruit			-					

PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

#### 1.3. Post-harvest Procedures

A single untreated and duplicate treated samples of blueberries and raspberries were harvested from each field trial. Specific harvesting procedures were not described; however, each sample weighed ≥1.1 lbs. Additional samples of raspberries were collected from the NY trial (Yates County) at various time intervals for residue decline samples. Samples were bagged and stored frozen (temperature not specified) on the day of harvest. Samples were shipped frozen within 4-28 days of harvest to BASF Agricultural Products Center (Research Triangle Park, NC) for analysis.

Table 1.3.1. Summar	y of Storage Conditio	TIS .	
Matrix	RAC or Extract	Storage Temperature ('C) (Analytical Laboratory)	Duration
Blueberry, highbush	Ветту	<-10	61-96 days (2.0-3.2 months)
Raspberry, red	Вепу	<-10	70-83 days (2.3-2.7 months)

# 1.4. Analytical Methods

Samples of blueberries and raspberries were analyzed for residues of BAS 510 F using LC/MS/MS method D9908, the data collection method for plant commodities. Briefly, blueberry and raspberry samples were extracted with methanol:water (70:30, v:v) and filtered. An aliquot of the filtrate was cleaned-up using C18 solid phase extraction. Residues were eluted with dichloromethane. The eluate was evaporated and residues were redissolved in ammonium formate:formic acid for analysis by LC/MS/MS; refer to the DER for MRID 45405027 for a complete description of the quantitation procedures. The limit of detection (LOD) was 0.025 ppm, and the validated limit of quantitation (LOQ) was 0.05 ppm for the residues of BAS 510 F in/on blueberries and raspberries. The concurrent recoveries obtained are presented in Table 2.1.

#### 2. Results

Table 2.1. Summary o	f Concurrent Analytical Method Va	lidation.	
Crop Matrix	Fortification Level (ppm)	Recoveries (%)	Mean Recovery
Blueberry, highbush	0.05, 5.0	94, 104	99
Raspberry, red	0.05, 5.0	91, 107	99

Submission # 2001-1027, 1036, 1043

Location (County, State, Year)	Crop Variety	Commodity	Formulation	Total Rate (lbs ai/A) [kg ai/ha]	PHI (days)	BAS 510 F residues (ppm)
	S.,	Blueber	ry, highbush			
Pience, WI, 1999	Blue Chop	Веггу	70% WG	1.48 [1.66]	0	0.923, 1.395
Benton, OR, 1999	Blue Crop	Berry	70% WG	1.50 [1.68]	o	0.486, 1.194
Yates, NY, 1999	Blue Ray and Blue Crop	Ветту	70% WG	1.52 [1.70]	Ò	1.142, 1.402
Jackson, WI, 1999	Berkley	Венту	70% WG	1.51 [1.69]	0	1.062, 1.460
Tift, GA, 1999	Tift Blue	Berry	70% WG	1.475 [1.65]	O	1.393, 1.522
Colquitt, GA, 1999	Climax	Berry	70% WG	1.498 [1.68]	0	2.171, 2.503
		Rasp	berry, red			
Yates, NY, 1999	Titau	Ветту	70% WG	1.51 [1.69]	0	2.064, 3.308
(decline study)		*			2	2.110, 2.529
					4	1.562, 2.288
					6	1.139, 2.024
					8	0.956, 1.525
Washington, OR, 1999	Mceker	Berry	70% WG	1.4996[1.68]	0	1.388, 1.589
Washington, OR, 1999	Tulamene	Berry	70% WG	1.4885[1.68]	0	1.565, 2.431

Commodity	Total Applic. Rate	PHI (days)		Resid	ue Levels (p	opm)	
	(lb ai/A) [kg ai/ha]		Minimum	Maximum	HAFT	Mean (median)	Std. Dev.
Blueberry, highbush	1.475-1.52 [1.65-1.70]	0	0.486	2.503	2.337	1.388 (1.394)	0.532
Raspberry, red	1,4885-1.51 [1.67-1.69]	0	1.388	3.308	2.686	2.058 (1.826)	0.723

## 3. Discussion

## 3.1. Methods

Blueberries and raspberries were harvested on the day (0-day PHI) of the last of four foliar spray applications of the 70% WG formulation at ~0.37 lb ai/A/application (0.41 kg ai/ha/application), with a 6- to 9-day retreatment interval, for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha).

Applications were made using ground equipment in a spray volume of 49.6-102.02 gal/A (555-1144 l/ha) of water with a spray adjuvant added. In one trial (Yates County, NY), additional raspberry samples were collected at 2, 4, 6, and 8 days following treatment to evaluate residue decline. We note that the 70% BAS 510 F WG formulation used in the field trials also contained another experimental active ingredient (BAS 500 F; pyraclostrobin) as part of the tank-mix; data for the BAS 500 F active ingredient were submitted separately.

Six blueberry trials were conducted in Regions 1 (1 trial), 2 (2 trials), 5 (2 trials), and 12 (1 trial) and three raspberry trials were conducted in Regions 1 (1 trial) and 12 (2 trials). The number and location of field trials conducted for blueberries and raspberries, as the representative crops of the berry group, are in accordance with the US EPA's guidance requirements (OPPTS 860.1500, Tables 2, 5, and 6). For the PMRA, the number and location of the submitted trials (see Table B.1.2) does not match Canadian guideline requirements (Dir 98-02).

During the course of the field trials, rainfall was reported as normal in the NY and OR trials; below normal in the GA trials; and above normal in the WI trials. Temperatures were reported as above normal in the WI and one of the GA trials, and normal in all the other trials. There was no irrigation in three (NY-1, WI-1, GA-1) of the trials.

Residues of BAS 510 F in/on blueberries and raspberries were quantitated using a concurrently validated LC/MS/MS method (D9908), the data collection method for plant commodities.

Maximum storage intervals of crop samples from harvest to analysis were 96 days (3.2 months) for blueberries and 83 days (2.7 months) for raspberries. Adequate storage stability data (refer to the DER for MRID 45405109) are available in five diverse matrices to support the storage conditions and intervals of samples from the submitted berry field trials.

## 3.2. Results

In <u>blueberries</u>, residues of BAS 510 F were 0.486-2.503 ppm in/on samples harvested on the day of the last of four foliar spray applications of the 70% WG formulation at 0.364-0.39 lb ai/A/application (0.407-0.437 kg ai/ha/application), for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha). Apparent residues of BAS 510 F were less than the method LOQ (<0.05 ppm) in/on six samples of untreated blueberries.

In <u>raspberries</u>, residues of BAS 510 F were 1.388-3.308 ppm in/on samples harvested on the day of the last of four foliar spray applications of the 70% WG formulation at 0.3653-0.39 lb ai/A/application (0.408-0.437 kg ai/ha/application), for a total rate of 1.49-1.51 lb ai/A (1.67-1.69 kg ai/ha). Apparent residues of BAS 510 F were less than the method LOQ (<0.05 ppm) in/on three samples of untreated raspberries. The residue decline data generated in New York State for raspberries indicated that residues of BAS 510 F decreased in a linear fashion. The residues decline data fits the linear equation y=-0.1815x + 2.6763 with a correlation coefficient (r2) of 0.9991. The equation predicts a half life of approximately 7 days and a complete dissipation of the residue by approximately 15 days.

Magnitude of the Residue OPPTS 860.1500 DACO 7.4.1 PC Code: 128008 MRID: 45405118 Submission # 2001-1027, 1036, 1043

# 4. Deficiencies

None for US registration. Additional field trials are required by PMRA for Canadian registration.

## 5. References

None